Canaigre Investigations*

I. A Review and Preliminary Report†

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Introduction

Tanning materials are essential for the production of vegetable tanned leathers. In normal times tannins equivalent to about 450,000 tons of 25 per cent tannin extract and valued at 15 to 20 million dollars have been used annually in the United States.

As a result of the present global war the demand for leather has been greatly increased, and deliveries of some of the imported tanning materials have been seriously threatened or entirely cut off. For a number of years this country has imported nearly two thirds of the vegetable tannins used in the manufacture of leather. About 90 per cent of the domestic supply△ has come from American chestnut wood.

Most of the commercial stands of this tree have been killed by the chestnut blight; chestnut extract is now produced almost entirely from dead wood. For several years quebracho, imported from South America either as wood or extract, has furnished about 40 per cent of the tannin used for making

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†This is the first of a series of papers reporting various phases of cooperative investigations of canaigre as a source of tannin by the Bureau of Agricultural and Industrial Chemistry and the Bureau of Plant Industry, Soils, and Agricultural Engineering, Agricultural Research Administration, United States Department of Agriculture, and the Texas Agricultural Experiment Station.

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△Data on the oak and hemlock barks produced domestically and leached and used directly in tannery liquors are not available. Therefore, figures for domestic production and consumption do not include tannin used in this way.

leather. Accessible stands of this material are rapidly becoming depleted[1], and establishment of plantations will be necessary if the use of this material is to be continued. Although complete figures on production of tannin from oak, hemlock and sumac are not available, the amount obtained from these sources is probably not much more than 10 per cent of the total used. These facts all emphasize the urgent need for the development of additional domestic sources of vegetable tannins. Several materials worthy of study have been suggested[2].

Research Program

Frey and Sievers[2] have outlined briefly the cooperative program of research on canaigre undertaken by the Bureau of Agricultural Chemistry and Engineering* and the Bureau of Plant Industry†. The work is divided into two general fields. One deals with the production of canaigre as a farm crop; the other deals with the processing of canaigre roots for the production of tanning extract and with the utilization of coproducts.

Successful growing of canaigre calls for thorough study of the various agronomic factors. This phase of the work, which is being conducted by the Bureau of Plant Industry, Soils and Agricultural Engineering, comprises selection of locations having suitable soil and climate, selection of suitable strains of planting stock, studies of propagation methods, determination of optimum spacing, fertilizer, and moisture requirements, and development of the best practices for cultivation and harvesting. Studies of yields, to determine whether they will justify production of canaigre as a farm crop, and of procedures for washing, cutting and drying the roots are also included.

Numerous laboratory and pilot-scale studies are necessary before canaigre can be adopted by the tanning industry as a useful and economical source of tannin. Some of these are now being conducted by the Bureau of Agricultural and Industrial Chemistry at the Eastern Regional Research Laboratory. They include investigations to determine the tannin content of canaigre roots from different sources and from various strains obtained from successive annual plantings, and effects on tannin content of various agronomic factors and cutting and drying procedures. The principal objectives, however, are the development of processing methods for canaigre roots by which tanning extracts of high quality can be produced and their value demonstrated. Pilot-scale tests will be conducted to determine the effectiveness of various canaigre preparations in tanning both heavy and light leathers. Consideration will be given to the economic utilization of valuable canaigre coproducts. This paper gives a brief review of the early work on canaigre and the results of some of the preliminary studies.

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*Now the Bureau of Agricultural and Industrial Chemistry.
†Now the Bureau of Plant Industry, Soils and Agricultural Engineering.

History

The Indians and the Mexicans have used canaigre, *Rumex hymenosepalus* Torr. for tanning for centuries[3]. The early missionaries called the plant sour cane, *Cana agria*. Records indicate that the first botanical specimens were collected by Thurber and sent to Torrey[4], who described the plant in 1859 and gave to it the name, *Rumex hymenosepalus*.

Between 1868 and 1905 considerable work was done on the development of canaigre as a commercial source of tannin. Results of the first analytical studies were published in 1876 by Voelcker[5], who found that a sample of "Raiz del Indico," later identified as canaigre root[6], contained 23.16 per cent tannin. Two years later, 1878, the United States Commissioner of Agriculture[7] published the results of analyses of two samples of roots from Texas. They contained 26.2 and 26.6 per cent tannin; also sugars and starch. In the report of the United States Commissioner of Agriculture for 1879, Saunders[8] published a colored lithographic plate showing canaigre in bloom and discussed its botanical identity.

During the next thirty years (1880-1910) canaigre received much attention. More than 50 publications appeared dealing with its growing and harvesting, its use in making tanning extract, and its probable future as a tanning agent. The most extensive studies recorded were those conducted by the State Agricultural Experiment Stations of Arizona, New Mexico, Texas, and California under the authorships of Collingwood, Toumey and Gulley[9]; Forbes[10]; Blount[11]; Hare[12]; Harrington and Adriance[13]; Bonner[14]; Hilgard[15]; and Colby[16]. Other important publications issued during this period were those of Trimble[17], Gulley[18], Eitner[19], and Knight[20].

Wild roots, which grew profusely in some areas of Arizona, New Mexico and Texas, were harvested and used for tanning locally. Several shipments of roots were made to Europe. Because of their susceptibility to fermentation and spoilage, whole roots proved to be unsatisfactory for shipping. Sliced, air-dried roots, however, were handled successfully. The experiment station reports dealt in considerable detail with botanical characteristics, geographic distribution, description of the plant and roots, tannin content, cultural aspects, harvesting, and the production of tanning extract.

Trimble[17] reported that canaigre roots were exhibited at the New Orleans Exposition in 1885-6 as a new tanning material, that ground canaigre roots were used in a number of tanneries, and that in tanning properties canaigre resembled gambier. Gulley[18] stated that in 1887 the first carload shipments of canaigre roots were made from Tucson, Arizona, to a seaport for export to Glasgow. From 1890 to 1892 shipments of several thousand tons of sliced, dried, sacked roots were made to Great Britain and Germany. A factory for making extract was established at Deming, New Mexico, in 1892, but the attempt to cultivate canaigre roots without irrigation was unsuccessful. The first extract placed on the market contained 30 per cent tannin. Later by

further concentration a thick extract containing 40-45 per cent tannin was prepared. This was of such consistency that it solidified when cool and could be shipped in wooden boxes. At the World's Fair in 1893 the Arizona Agricultural Experiment Station exhibited canaigre roots as dug, sliced, grated, and dried, and also extracts containing 30 to 45 and 55 to 60 per cent tannin (solid). Eitner[19] after conducting tanning experiments concluded that canaigre was suited for tanning upper, saddle and fancy leathers. He found that it tanned quickly and produced a soft, strong leather. It has also been used successfully with pine bark for tanning heavy leather. Knight[20] reported that experimental plantings of canaigre at Mildura, Australia, gave results that compared favorably with those obtained in the United States.

In the present century there have been occasional periods of revived interest in canaigre. Fifteen or more publications have appeared. The more important of these are listed [21–27].

So far as we know there is at present no established market for canaigre, and its use, if any, is limited to small-scale or home-tanning projects among Mexicans or the inhabitants of the southwestern United States. In the opinion of those who have worked with it, however, canaigre possesses the properties of a valuable tanning material that can be used in tanning both light and heavy leathers.

Failure of canaigre to find an established place among accepted commercial tanning materials may have been due to such causes as a plentiful supply of inexpensive oak and hemlock barks, production of high-quality tanning extract from chestnut wood, and importation of quebracho at acceptable prices. In the nineties truck transportation facilities and good roads were not generally available. When the wild canaigre supply near railroad shipping points was exhausted, transportation became a problem. Failure to cultivate canaigre as a crop may have been due to lack of irrigation facilities. Knowledge concerning leaching and producing tanning extracts in commercial quantities was limited, and little or no thought was given to utilization of coproducts. The starch in canaigre roots made leaching at high temperature difficult, and the sugars, which were leached out with the tannins, lowered the purity of the tanning extracts.

Today the picture is changed. Available supplies of domestic tanning materials are dwindling. Transportation facilities are greatly expanded and improved. Much experience and technical knowledge have been acquired thus providing workers with the training necessary to cope with problems of manufacture and utilization of coproducts. Rapid development of a domestic vegetable tannin supply other than the undeveloped barks and woods of our forests depends on the production of an annual or quick-growing crop. Canaigre possesses a distinct advantage, since it can be produced in one growing season. It would be another product for the farmer, making possible greater diversification of crops and increased land utilization. The quantities grown

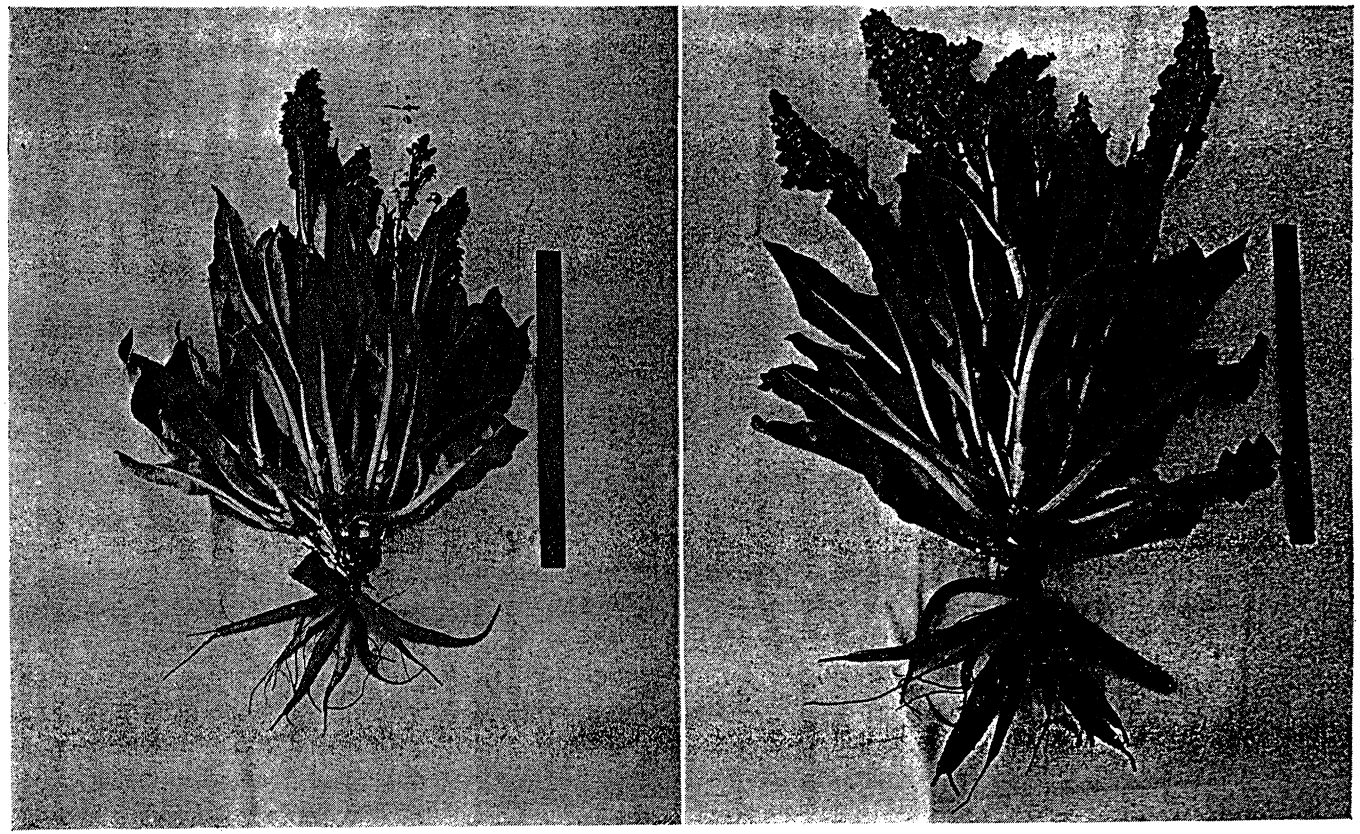

FIGURE I—Canaigre Plants
Flowers, leaves and roots on a one-year-old plant (left) and a two-year-old plant (right).
The parent root crown is noticeable among roots of one-year-old plant.

and made available to the tanning industry could be regulated to meet changing demands. The apprehension that domestic supplies might become exhausted with no replacements in sight would be removed.

The Plant

Canaigre grows naturally in Texas, New Mexico, Arizona, Southern California, and in other sections of the southwestern part of the United States and northern Mexico. It flourishes along the Rio Grande and other river bottoms and grows well on the mesas.

In some respects, the canaigre plant (Fig. I)* resembles other members of the dock family. It grows from one to three feet high, and in some cases the large succulent leaves have a spread of more than 18 inches. Growth usually begins soon after the fall or early winter rains, but in unirrigated land when rainfall is low it may not begin until February or March. The plant matures and blooms in the spring and then, usually in May or June, withers and dies back to the ground. The tuberous roots (Fig. II), which are somewhat similar to those of the sweet potato or dahlia, vary considerably in general appearance, weight and number produced per hill. The weight of single roots may range from a few ounces to over a pound. In some cases more than 20 roots have been produced in a hill in one year, and individual hill yields of more than 6 pounds have been recorded. The roots may be harvested at any time after the plant dies back to the ground, but it is claimed that the tannin content increases during the dormant summer period; it is therefore advantageous to defer harvest until time for replanting, which is usually in late August or early September.

Freshly dug canaigre roots contain different amounts of moisture. In general, however, the average moisture content ranges from 65 to 75 per cent. The tannin content also varies with strains and apparently with different localities. Wild roots, two or more years old, generally contain from 20 to 30 per cent tannin on a moisture-free basis. Some samples have had more than 35 per cent. One-year-old cultivated roots usually are somewhat poorer in tannin than older wild roots.

Preliminary Studies

As pointed out before, the data in this paper are presented simply as an introduction to various phases of studies which will be discussed in detail in later papers. For determination of the tannin content of wild roots, samples have been secured from several sources. In some cases these were composites of several hills†; in others they represented single isolated hills. For this

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†The term "hill" indicates roots produced by a single plant.

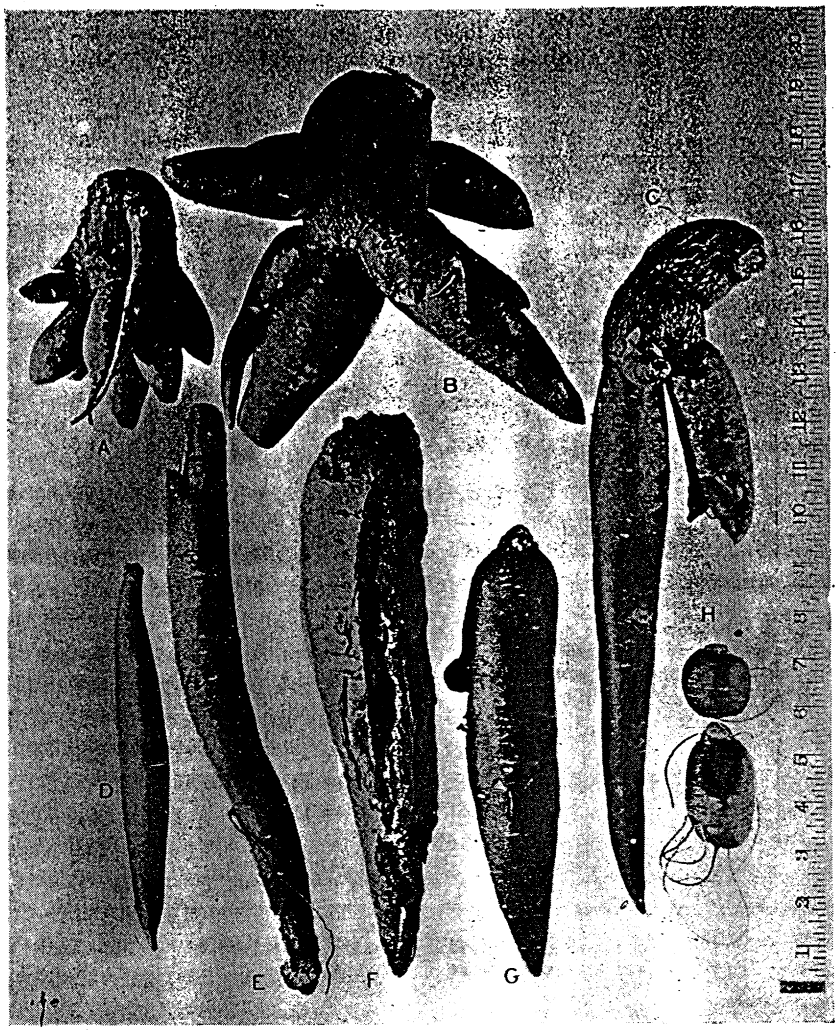

FIGURE II—Canaigre Roots

A and B, branching types; C, new growth on parent root; D and E, long slender roots; F, large typical root cracked open and showing decay at crown; G, average normal root; and H, globular-shaped roots.

reason they may not be truly representative of the area from which they came. Results of analyses of nine such samples collected in California, Arizona and New Mexico are recorded in Table I. With the exception of two samples, one from Albuquerque, New Mexico, and the other from Wenden, Arizona, these agree in tannin content, and confirm previously published data. With

TABLE I

Analyses of Canaigre Roots Collected from Wild Plants in Arizona, California and New Mexico

(Results on moisture-free basis)

Source	Collection Date	Total Solids	Soluble Solids	Non Tannins	Tannin	pH*	Purity†
		%	%	%	%		
Rillito bottoms near Tucson, Arizona‡	5/24/37	55.2	52.2	22.6	29.6	5.2	56.7
Near Dragoon, Arizona (altitude. 4000 feet‡)	5/25/37	53.5	51.8	21.4	30.4	5.6	58.7
Santa Paula, California‡	5/27/37	52.1	50.8	25.1	25.7	5.5	50.6
Rio Grande bottoms, 15 miles from Albuquerque, New Mexico§	5/28/37	42.0	40.5	31.1	9.4	5.1	23.2
Near Dragoon, Arizona (altitude 4000 feet)	5/25/37	56.1	54.8	22.8	32.0	5.7	58.3
State College, New Mexico	6/ 9/37	45.3	44.7	22.3	22.4	4.5	50.1
Shiprock, New Mexico (altitude 5000 feet)	8/18/37	54.3	52.9	32.1	20.8	5.6	39.3
Wenden, Arizona, 40 miles West of Aquilla	8/ 2/43	43.3	41.2	22.7	18.5	...	44.9
Sacaton, Arizona	9/11/42	55.0	52.9	22.3	30.6	..	57.8

*pH was determined on the water extract.

†Purity = tannin/soluble solids x 100.

‡The interior of roots was light pink to almost white.

§The interior of the roots was yellow.

TABLE II

Analyses of Canaigre Roots Collected from Wild Plants near Big Spring, Texas, April, 1937

(Results on moisture-free basis)

Source	Sample No.	Description	Total Solids	Soluble Solids	Non Tannins	Tannin	pH*	Purity†
			%	%	%	%		
Billings Farm‡	1	Not current year's growth	51.0	49.1	29.9	19.2	5.2	39.1
	2	Sliced and air dried before shipment§	37.5	36.5	19.5	17.0	5.6	46.6
	3	Current year's growth	43.1	42.3	33.3	9.0	5.6	21.2
	4	Dead roots	17.8	17.0	7.4	9.6	5.8	56.4
	5	Blown out of soil by wind	58.6	56.7	33.4	23.3	5.7	41.1
Cantrell Farm‡	1	Not current year's growth	45.4	43.1	27.6	15.5	5.7	35.9
	2	Sliced and air dried before shipment§	32.5	32.2	18.0	14.2	5.7	44.1
	3	Current year's growth	51.3	50.5	35.0	15.5	5.6	30.6
Nelson Farm‡	1	Not current year's growth	41.8	40.2	26.3	13.9	5.7	34.6
	2	Sliced and air dried before shipment§	28.6	27.5	16.0	11.5	5.7	41.8
	3	Current year's growth	54.6	53.8	38.6	15.2	5.6	28.2

*pH was determined on the water extract.

†Purity = tannin/soluble solids x 100.

‡In vicinity of Big Spring, Texas.

§This sample was the same as the one immediately preceding except that it was sliced and dried in th field before shipment.

the exception mentioned, the tannin content of the Arizona samples was between 29.6 and 32.0 per cent. That of samples from New Mexico was lower.

Analyses of a series of samples collected near Big Spring, Texas, in 1937 are recorded in Table II. These are of interest because of the consistent differences between samples sliced and dried at the laboratory and those sliced and dried in the field before shipment. The latter were much thicker than the former, and for this reason probably dried much more slowly. They were consistently lower in total extractives, non tannins and tannins than the samples prepared in the laboratory. This loss in water solubles may have occurred as a result of fermentation, or of enzyme action and fermentation. Determinations of sugars on these samples were not made, however, so no confirmation of this suggested explanation was obtained at that time.

TABLE III

ANALYSES OF CANAIGRE ROOTS COLLECTED FROM 25 WILD PLANTS ON THE MESA 23 MILES NORTH OF LAS CRUCES, NEW MEXICO, SEPTEMBER, 1937

(Results on moisture-free basis)

Plant No.	Total Solids	Soluble Solids	Non Tannins	Tannin*	Purity	Reducing Sugars	Total Sugars†
	%	%	%	%		%	%
1	30.1	28.9	17.7	11.2	38.7	7.8	14.6
2	38.8	35.8	21.3	14.5	40.5	4.1	10.9
3	37.0	34.4	15.9	18.5	53.7	2.6	8.6
4	38.2	34.4	14.5	19.9	57.8	4.1	8.1
5	45.5	42.8	22.6	20.2	47.2	4.8	13.9
6	47.0	43.3	21.8	21.5	49.6	6.6	10.5
7	49.8	42.7	20.6	22.1	51.7	9.7	10.7
8	43.7	39.9	17.8	22.1	55.4	5.6	10.4
9	48.5	45.5	23.2	22.3	49.0	6.6	14.3
10	43.2	41.8	19.4	22.4	53.6	3.1	10.3
11	43.6	40.2	17.6	22.6	56.2	4.5	13.2
12	55.7	52.0	28.7	23.3	44.8	8.6	19.6
13	51.3	46.9	23.5	23.4	49.9	....	....
14	43.9	39.4	15.3	24.1	61.1	2.9	8.6
15	47.9	45.7	21.1	24.6	53.8	9.6	12.5
16	48.2	47.2	21.1	26.1	55.3	4.6	8.1
17	46.7	44.2	17.7	26.5	59.9	6.3	10.2
18	53.2	50.6	23.0	27.6	54.5	9.2	13.5
19	56.6	55.6	27.6	28.0	50.4	15.8	16.8
20	53.2	51.5	23.2	28.3	54.9	10.8	14.4
21	54.6	49.5	21.0	28.5	57.6	6.6	12.7
22	53.8	51.0	21.9	29.1	57.1	7.0	10.8
23	52.0	49.4	18.6	30.8	62.3	4.0	11.6
24	59.5	57.1	23.6	33.5	58.7	8.3	14.9
25	58.9	55.7	20.4	35.3	63.3	6.7	12.4

*Average tannin content, 24.3 per cent.

†Average total sugars, 12.1 per cent.

To furnish planting stock for the cultural studies, which will be reported in later papers, wild roots were dug on the mesa 23 miles north of Las Cruces, New Mexico. The normal range in composition that may be expected in such roots is shown in Table III. Each sample was a composite of the roots from a single plant after two roots had been removed for planting stock. Each cluster contained roots of different but unknown ages. Statements in the literature indicate that roots more than one season old are usually richer in tannin than those matured in a single season. The tannin content of most of the samples represented in Table III was somewhat higher than may generally be expected in cultivated roots one year old. On the whole, the twenty-five samples present a fair picture of the range in amounts of tannin and sugar in wild roots produced in this area. Tannin ranged from 11.2 to 35.3 per cent, with an average of 24.3, and the total sugars ranged from 8.1 to 19.6 per cent, with an average of 12.1. Systematic analyses of roots for determination of starch have not been made, but more than 26 per cent of crude starch has been separated mechanically from one sample of roots. Analysis of another sample of cultivated roots showed 18.4 per cent of total sugars (after inversion) and 12.6 per cent of starch. The starch content varied in different lots of roots, but its range has not been determined.

For the selection of strains high in tannin, a knowledge of the tannin content of the planting stock is essential. In some cases whole roots are planted; in

TABLE IV

VARIATION IN THE TANNIN CONTENT OF ROOTS PRODUCED BY FIVE CULTIVATED CANAIGRE PLANTS DURING ONE SEASON'S GROWTH

(Results on moisture-free basis)

Plant No.	Root	Total Solids	Soluble Solids	Non Tannins	Tannin*	Purity	pH
		%	%	%	%		
1	a	52.5	50.7	36.9	13.8	27.2	5.7
1	b	55.7	53.7	38.3	15.4	28.7	4.2
1	c	52.1	50.8	36.6	14.2	27.9	4.7
2	a	55.6	53.4	33.8	19.6	36.7	4.7
2	b	50.6	49.6	31.6	18.0	36.3	4.7
2	c	50.9	49.5	32.0	17.5	35.3	4.7
3	a	61.7	60.2	36.7	23.5	39.0	4.9
3	b	63.3	61.4	37.6	23.8	38.8	4.7
3	c	66.3	58.7	38.0	20.7	35.3	4.6
4	a	59.8	56.9	36.4	20.5	36.0	4.8
4	b	56.0	53.8	34.9	18.9	35.1	5.7
4	c	58.0	56.5	35.1	21.4	37.9	5.5
5	a	54.5	52.9	31.7	21.2	40.1	5.8
5	b	56.4	55.0	33.2	21.8	39.6	5.8
5	c	53.8	52.8	31.4	21.4	40.5	5.9

*The greatest variation in tannin content from one plant (3 roots) was 3.1 per cent; the least variation one plant (3 roots) was 0.6 per cent.

others only the root crowns are used. When whole roots are planted, the most representative sample obtainable for checking tannin content of planting stock would be one from roots of the same age from the same hill. To obtain information concerning the variation in tannin that might be expected, analyses have been made of 15 individual roots, 3 from each of 5 planted hills, produced during one season's growth. The results (Table IV), although few in number, indicate that tannin in roots of the same age and from the same hill may vary as much as 3.1 per cent. In this test, however, the variation in tannin content between roots from the same hill was significantly less than the variation of the averages for different hills.

When root crowns are used for planting, a knowledge of the tannin content of the roots from which the crowns are obtained may be required. Analyses of the corresponding root bodies would appear to be the logical method of acquiring this information, providing there is no significant difference in tannin content between these parts. To check the relation between tannin content of crowns and bodies of the same roots, five fairly large roots were cut into crown and body portions, which were analyzed separately for tannin. The results of this study are shown in Table V. Although there are slightly higher tannin contents in the body portions of these roots than in the crowns, the differences are not significant and are definitely less than those between roots. Results of this test indicate that analyses of root bodies will determine whether crowns used as planting stock should be in the high-, medium-, or low-tannin class.

The review of the early work dealing with the growing and harvesting of canaigre and its utilization as a tanning material, together with the program outlined and the preliminary results presented in this paper, point to some of the agronomic and laboratory problems that must be worked out before

TABLE V

TANNIN CONTENT OF CROWNS AND BODIES FROM THE SAME CANAIGRE ROOTS

(Results on moisture-free basis)

Root Number	Part of Root	Total Solids	Soluble Solids	Non Tannin	Tannin	Purity
		%	%	%	%	
1	Crown	62.6	60.7	43.0	13.7	22.6
1	Body	66.3	65.6	48.6	17.0	25.9
2	Crown	56.9	55.1	37.0	18.1	32.8
2	Body	60.6	58.8	38.9	19.9	33.8
3	Crown	66.2	64.6	43.2	21.4	33.1
3	Body	67.5	65.3	43.8	21.5	33.0
4	Crown	62.2	59.5	48.4	11.1	18.7
4	Body	61.6	59.6	47.3	12.3	20.6
5	Crown	56.5	53.9	37.2	16.7	31.0
5	Body	60.8	57.7	40.4	17.3	30.0

canaigre can be successfully developed as an economic source of tannin. Field and laboratory studies on these problems are now in progress.

Summary

The program for cooperative investigation of canaigre as a source of tannin is outlined, and the early history of canaigre is briefly reviewed. The canaigre plant and roots are described, and its habitat is given. Data are presented showing (1) that samples of wild roots collected from twelve locations in four States ranged in tannin content from 9.0 to 32.1 per cent; (2) that in 25 samples collected in one area the tannin content ranged from 11.2 to 35.3 per cent and the total sugars from 8.1 to 19.6 per cent; (3) that the tannin contents of roots of the same age and from the same hill varied as much as 3.1 per cent, which was significantly less than the variation of averages representing different hills; and (4) that analyses of bodies of roots can be used to determine whether the corresponding crown portions, taken for planting, represent strains of high-, medium- or low-tannin content.

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